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In Situ Dehydration Experiments of NH4-RUB-29
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Introduction: Based upon previous ammonium exchange experiments of as synthesized RUB-29 and Na-RUB-29 [1] to conduct NH_4-RUB-29 material, we have further investigated this to prepare H-form of NH_4-RUB-29 (hereafter designated H-RUB-29). In terms of catalysts, H-RUB-29 belongs to a new type of catalysts because H-RUB-29 possesses a new type of acid center involving HO-Li-O-Si configuration. The resulting catalytic properties may be very different from that we observed in aluminosilicate catalyst materials. In the first step to the investigation of H-RUB-29, we carried out time-resolved dehydration experiments of NH_4-RUB-29 to prepare thermally and chemically stable H-form of RUB-29.
Methods and Materials: A sample of NH_4-RUB-29 was prepared ex situ with as-synthesized RUB-29 by in the presence of a 0.5M NH_4NO_3 solution. The NH_4-RUB-29 sample was filled into a thin quartz capillary (0.7mm diameter) and then heated continuously with a heating rate of 10°C/min. The time-resolved dehydration of NH_4-RUB-29 was detected using synchrotron X-ray powder diffraction techniques with the translating imaging plate in a temperature range between 25°C and 800°C. To see changes in cell parameters due to dehydration and activation with increasing temperature, four different diffraction profiles obtained at room temperature (hydrated NH_4-RUB-29), 180°C (dehydrated NH_4-RUB-29), and 400°C (H-RUB-29) were chosen and analyzed with the Rietveld program GSAS [2].
Results: As shown in Fig. 1, the dehydration of NH_4-RUB-29 material finished at about 150°C and the transformation of dehydrated NH_4-RUB-29 to H-RUB occurred continuously between 150°C and 500°C. Till the H-RUB-29 disappeared, the material maintained the original space group symmetry I222 for as-synthesized RUB-29. The lattice parameters of hydrated, dehydrated NH_4-RUB-29, and H-RUB-29 are given in Table 1.

	hydrated NH_4-RUB-29 at RT	dehydrated NH_4-RUB-29 at 180°C	H-RUB-29 at 400°C
a [Å]	11.029(1)	11.038(1)	11.035(1)
b [Å]	17.269(2)	17.269(1)	17.272(2)
c [Å]	24.009(3)	23.935(2)	23.983(2)
V [$Å^3$]	4572(1)	4562(1)	4571(1)

After the dehydration stage, the peak intensity of the reflection (110) at 3.75°(2θ) increased continuously and reached the maximum at 400°C. In contrast, the peak intensities of the other reflections such as (002) did not or the changed was less between 150°C and 400°C. There was no discontinuity at peak positions of all reflections after the dehydration at 150°C up to 600°C. Based on these observations, we concluded that the activation of NH_4-RUB-29 to H-RUB-29 was finished at about 400°C without changing of the space group symmetry I222.
As shown Fig. 1, H-RUB-29 can be structurally stable up to 600°C where a phase transition to an unknown high temperature phase occurred. A similarity to this phase transition was also observed during the dehydration of as-synthesized RUB-29 above 650°C. Therefore, we assume this phase can be considered as the high temperature modification of the RUB-29 structure.
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References:
[1] M. Kleinsorge, S.-H. Park, J.B. Parise (SUNY, Stony Brook) and J.C. Hanson (BNL), "Time-resolved ion-exchange experiments of microporous lithosilicate RUB-29", BNL abstract No.Klei552, 2001
[2] A.C. Larson, B. Von Dreele, GSAS: General Structure Analysis System, Los Alamos National Laboratory, Los Alamos, New Mexico 87545, USA

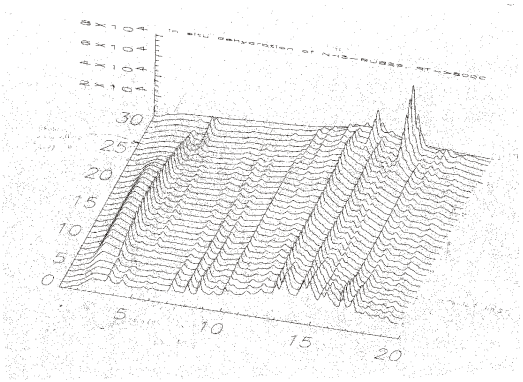

Fig.1.Time-resolved synchrotron X-ray diffraction pattern observed during the heating process from 25°C to 800°C of NH_4-RUB-29